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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 13255.8-2009

Replacing GB/T 13255.8-1991

**Test methods of caprolactam for industrial use -- Part 8:
Determination of cyclohexanone oxime content**

工业用己内酰胺试验方法 第8部分：环己酮肟含量的测定

(English Translation)

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Foreword

GB/T 13255 "Industrial caprolactam test method" is divided into eight sections.

- Part 1. Determination of a 50% aqueous solution of chromaticity spectrophotometry;
- Part 2. Determination of crystallizing point;
- Part 3. Determination of permanganate absorption spectrophotometry value;
- Part 4. After distillation of volatile alkali content of titration;
- Part 5. 290nm wavelength measured absorbance;
- Part 6. Determination of acidity or alkalinity;
- Part 7. Determination of iron content;
- Part 8. Determination of cyclohexanone oxime content.

This section GB/T 13255 Part 8.

The partial modification of the Russian Federation adopted standards GOCT26743.6-1985 "Determination of caprolactam cyclohexanone oxime content." According to this section GOCT26743.6-1985 redrafted. The standard lists the reg number in Appendix A and GOCT26743.6-1985 reg control list numbered.

Taking into account China's national conditions, in this section when using GOCT26743.6-1985 made some editorial changes, this portion the main technical difference GOCT26743.6-1985 follows.

Preparation --- cyclohexanone oxime different standard solution, dissolved in water directly GOCT26743.6-1985, this section is to use a few drops it was dissolved in ethanol and then diluted with water;

--- Results of different expressions, GOCT6743.6-1985 percentage by mass expressed by this section in mg/kg.

This Part replaces GB/T 13255.8-1991 "Determination industrial caprolactam cyclohexanone oxime content." this section compared with the GB/T 13255.8-1991 The main changes are as follows.

--- Increasing the level of balance requirements (see 5.3);

--- Will analyze the water level defined as "third grade water GB/T 6682-2008 specified in" (1991 Edition Chapter 3 edition Chapters 4);

--- The microburette to different specifications of the Class A Graduated pipettes (1991 edition, Chapter 4, Chapter 5 of this edition);

--- Representation of the results of cyclohexanone oxime content from "the mass fraction (10-6)" to "mg/kg" (1991 version of Chapter 6 of the present Chapter 7 edition);

--- The "poor results of two parallel determination of not more than 10×10^{-6} " to "difference between two parallel determination results is not more than 5mg/kg" (1991 edition, Chapter 6, Section 7 of this edition);

--- Added "Report" (see Chapter 8).

Appendix A of this section is an informative annex.

This part is proposed by the China Petroleum and Chemical Industry Association.

This part of the National Standardization Technical Committee on Organic Chemistry Club (SAC/TC63/SC2) centralized.

This section is drafted. DSM Nanjing Chemical Co., Ltd.

1 Scope

This section GB/T 13255 specifies the method for industrial use - Determination of cyclohexanone oxime caprolactam content of hydroxamic acid iron spectrophotometer DEGREE.

2 Normative references

The following documents contain provisions which, through reference GB/T 13255 in this section constitute provisions of this section. For dated reference documents Member, all subsequent amendments (not including errata content) or revisions do not apply to this section, however, encouraged to reach under this section Parties to research agreement to use the latest versions of these documents. For undated reference documents, the latest versions apply to this section.

GB/T 6682-2008 analytical laboratory use specifications and test methods (ISO 3696. 1987, MOD) GB/T 8170 repair value expressed about the rules and limit values
judgment

3 PRINCIPLE OF THE METHOD

Cyclohexanone oxime when hydrolysis in acidic medium of hydroxylamine with formaldehyde to generate formylation hydroxamic acid under conditions of ammonium persulfate (aka Carboxamido hydroxamic acid), formylation hydroxamic acid can form a red complex with ferric ion, the maximum absorption wavelength at 500nm with a spectrophotometer Measuring absorbance.

4 Reagents

Unless otherwise indicated in the analysis using only recognized as analytical reagents and third grade water GB/T 6682-2008 stipulated.

4.1

ammonium persulfate.

4.2

sulfuric acid solution. 1 + 90.

Formaldehyde solution 4.3. 5 + 1.

4.4

ammonium ferric sulfate $[\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ solution. 100g/L, weighed 10g ammonium ferric sulfate dissolved in water and diluted to 100mL, Then add 1.5mL solution of sulfuric acid (1 + 90) mix, when turbidity to be filtered.

4.5

cyclohexanone oxime. recrystallized twice from benzene to obtain.

Cyclohexanone oxime standard solution 4.6 Ring. 100mg/L, weighed 0.100g cyclohexanone oxime, to the nearest 0.001g, a 50mL beaker, number After dissolution drops of ethanol, water and quantitatively transferred to a 1000mL volumetric flask, and dilute to volume, and mix.

5 Instruments

5.1

Spectrophotometer. Accuracy $\pm 0.004A$. 5cm with absorption cell 2.

5.2

Graduated pipettes. A level, different specifications.

5.3

Balance. sensitivity, respectively 0.1g and 0.1mg.

6 Step, Analysis

Draw 6.1 Working curve with graduated pipettes are accurate Pipette 0.0mL, 1.0mL, 2.0mL, 3.0mL, 4.0mL, 5.0mL standard solution of cyclohexanone oxime